DEPARTMENT OF THE ARMY
US ARMY CHEMICAL MATERIALS AGENCY
TOOELE CHEMICAL AGENT DISPOSAL FACILITY
11620 STARK ROAD
STOCKTON, UTAH 84071

REPLY TO
ATTENTION OF:

HAND DELIVERED

MAR 2 0 2008

UTAH DIVISION OF
SOLID & HAZARDOUS WASTE
08.01107

March 10, 2008

Tooele Chemical Agent Disposal Facility PM0103-08

SUBJECT: 4.2-inch Mustard (HT) Mortar Sampling and Analysis Plan

Mr. Dennis Downs, Director
Utah Department of Environmental Quality
Division of Solid and Hazardous Waste
288 North 1460 West
Salt Lake City, Utah 84116-0690

Dear Mr. Downs:

Enclosed is the TOCDF 4.2-inch Mustard (HT) Mortar Sampling and Analysis Plan, as required under paragraph 2.2.1.3.3 of Attachment 2 of the TOCDF RCRA Permit.

If you have any questions, please contact Ms. Elizabeth A. Lowes at (435) 833-7832 or Mr. T. Trace Salmon at (435) 833-7428.

Sincerely,

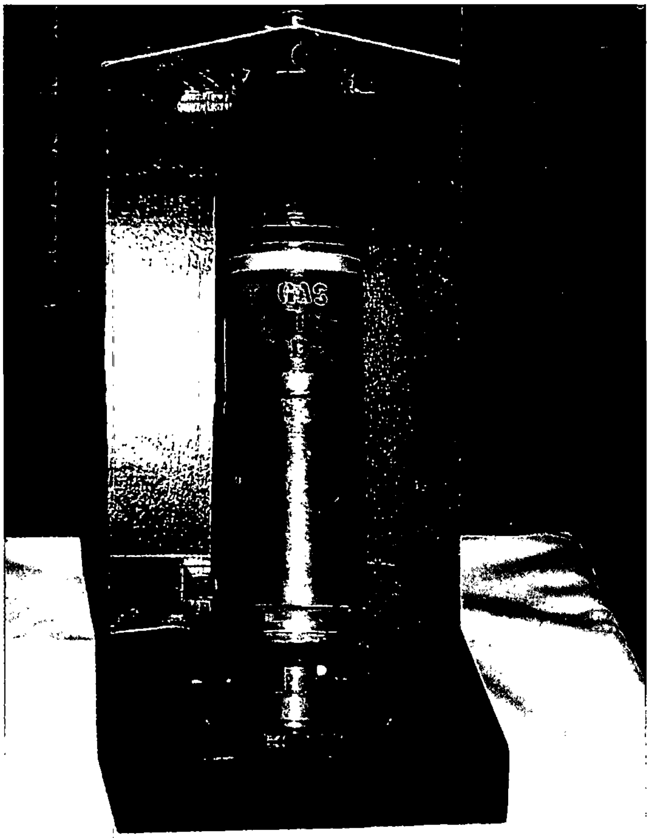
Gary W. McCloskey
EG&G Defense Materials, Inc.
*CERTIFICATION STATEMENT

Thaddeus A. Ryba, Jr.
TOCDF Site Project Manager
*CERTIFICATION STATEMENT

Enclosure

* I CERTIFY UNDER PENALTY OF LAW THAT THIS DOCUMENT AND ALL ATTACHMENTS WERE PREPARED UNDER MY DIRECTION OR SUPERVISION IN ACCORDANCE WITH A SYSTEM DESIGNED TO ASSURE THAT QUALIFIED PERSONNEL PROPERLY GATHER AND EVALUATE THE INFORMATION SUBMITTED BASED ON MY INQUIRY OF THE PERSON OR PERSONS WHO MANAGE THE SYSTEM, OR THOSE PERSONS DIRECTLY RESPONSIBLE FOR GATHERING THE INFORMATION, THE INFORMATION SUBMITTED IS, TO THE BEST OF MY KNOWLEDGE AND BELIEF, TRUE, ACCURATE AND COMPLETE. I AM AWARE THAT THERE ARE SIGNIFICANT PENALTIES FOR SUBMITTING FALSE INFORMATION, INCLUDING THE POSSIBILITY OF FINE AND IMPRISONMENT FOR KNOWING VIOLATIONS

TOOELE CHEMICAL AGENT DISPOSAL FACILITY (TOCDF)



4.2-inch Mustard (HT) Mortar Sampling and Analysis Plan

March 2008

EG&G Defense Materials, Inc.

Environmental Department

11600 Stark Rd.

Stockton, UT 84071

EG&G A Division of URS

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1.0 INTRODUCTION

The United States Army (Army), through the office of the Chemical Materials Agency (CMA), is responsible for ensuring the safe and timely destruction of obsolete chemical weapons presently stored on the Deseret Chemical Depot (DCD), located 20 miles south of Tooele, Utah. The demilitarization of the DCD chemical stockpile takes place at the Tooele Chemical Agent Disposal Facility (TOCDF), which is located within the boundaries of the DCD. This hazardous waste treatment facility was designed and built for the Army to thermally treat chemical Agents GB, VX, and mustard (H-series); drained munitions; contaminated refuse; bulk containers; liquid wastes; explosives; and propellants. EG&G Defense Materials, Inc., (EG&G) operates the TOCDF under contract to the Army through the office of the CMA.

This sampling and analysis (S&A) plan will be conducted to provide assurance that the TOCDF incinerators will be operated in compliance with the performance standards specified in Module VI of the Resource Conservation and Recovery Act (RCRA) Permit while processing 4.2-inch HT Mortars. The S&A Plan will be conducted in a manner that meets the requirements of the Waste Analysis Plan (WAP). This plan identifies the samples to be collected and the analyses to be performed to characterize 4.2-inch HT Mortars for organic compounds and metals.

The analyses methods are approved by the State of Utah Division of Solid and Hazardous Waste (DSHW) and are based on United States Environmental Protections Agency (EPA) SW-846 methods. (1)

1.1 4.2-inch HT MORTAR BACKGROUND

The DCD stockpile includes two types of 4.2-inch Mortars: those filled with Agent HT and those filled with Agent HD. This study is applicable to the 4.2-inch Mortars filled with Agent HT. The H in the HT stands for mustard; the chemical name for mustard is *bis*(2-chloroethyl) sulfide ($ClCH_2CH_2SCH_2CH_2Cl$). The T in the HT stands for *bis*(B-chloroethyl-thioethyl) ether [$O(CH_2CH_2SCH_2CH_2Cl)_2$].

The HT used to fill the DCD 4.2-inch Mortars was manufactured in Canada and England during the mid 1940s using what was known as the "60° Centigrade Process." Rather than creating HT by mixing mustard manufactured using the Levinstein process followed by the addition of T, HT was manufactured from a chemical process that, when completed, resulted in a mixture of H and T. The following describes this process (with references to two locations in England where the process was developed).

> At the Randle Works, HT 60/40 is prepared by much the same process as described....There is no change in the hydrochloric acid system. The thiodiglycol [$S(CH_2CH_2OH)_2$], 50 gallons, is preheated to 60 degrees centigrade and is

introduced into a reaction vessel together with about 11 ½ gallons of hydrochloric acid. Gaseous hydrogen chloride is bubbled through, rapidly at first, then at such a rate as to maintain a small exhaust of excess gas. The reaction mixture is maintained at about 110 degrees centigrade by the heat of the reaction. After 30 minutes, when the reaction is almost complete, the supply of hydrogen chloride is reduced and adjusted to maintain a small exhaust for 45 minutes longer. From this point on, the reaction mixture is treated as at Sutton Oak.... (2)

...After the reaction, the mixture is air-blown to remove some of the excess hydrogen chloride, and 25 gallons of water is added to dissolve the salt originally present as an impurity in the thiodiglycol. After further blowing, the batch is drawn to a settler, where the HT separates as the lower layer and the toxic acid as the upper layer. The HT layer is separated, allowed to settle longer, then dried under vacuum and stored. (2)

Table 1 shows that the resulting product typically contained the following concentrations of organic compounds.

Table 1: HT Organic Composition

	Chemical Name & Formula	Wt %
H	*bis* (2-chloroethyl) sulfide ($ClCH_2CH_2SCH_2CH_2Cl$)	60
T	*bis* [2-2-(chloroethylthio)ethyl]ether $O(CH_2CH_2SCH_2CH_2Cl)_2$	26
Q	Sesquimustard (1,2-bis(2-chloroethylthio)ethane) $ClC_2H_4SC_2H_4SC_2H_4Cl$	4
T1*	High molecular wt. homologues such as T1 $ClCH_2CH_2S(CH_2H_2COCH_2CH_2S)_nCH_2CH_2Cl$	10

*The n in T1 is equal to 2; n can equal 3, 4 and higher for other homologues

The 4.2-inch HT Mortars were filled at Pine Bluff Arsenal (PBA) in 1944. The PBA munitions manufacturing lot number generated by their filling designates mortars that reflect the same types of mortar casings, fuses, propellants, and other features. If a critical component was changed, a new lot number was issued. Note that the HT fill itself was not deemed a critical component, so agent lot numbers were not assigned by the Army. All the HT was identified as being equivalent because of its consistency.

The 4.2-inch HT manufacturing lot numbers, therefore, have no relation to HT agent manufacturing lot numbers. Further, because the HT mortars that are stored at DCD underwent two renovations, during which critical components were changed, the current DCD lot numbers include a mixture of mortars belonging to the original HT mortar manufacturing lot numbers that were assigned in the mid 1940s when the mortars were filled. Therefore, the population of DCD 4.2-inch HT Mortars is a random population in regards to the HT fill.

The content of the DCD HT mortar stockpile was characterized to some extent during a study conducted by Parsons Corporation for the purpose of developing the design of a Projectile Washout System (PWS). This PWS was intended for use at the Pueblo Chemical Agent Destruction Pilot Plant (PCAPP). During the testing, liquid HT samples were collected from both individual mortars and from a tank to which the HT fill of multiple mortars had been transferred. In the course of this test, some mortars were observed to contain a paste-like solid that remains in the mortars after the liquid HT has been drained. Three samples of the paste-like heel were collected and analyzed. The analytical results for the liquid HT and paste-like solid heel obtained during this testing are provided in Table 2.

Table 2: Parsons PWS Report HT Analysis Summary

Liquid HT Organic Compounds (wt.%)	Liquid HT[1,3]			Paste-Like Solid Heel[2,4]			Paste-Like Heel Organic Compounds (wt.%)
	Ave	Max	Min	Ave	Max	Min	
Bis (2-chloroethyl) sulfide (HD Agent)	56.5	60.1	52.3	0.84	1.6	0.44	1,4-Dithiane
bis [2-(2-chloroethylthio)ethyl] ether (T)	32.7	36.5	28.5	1.5	2.8	0.81	1,4-Thioxane
1,2-*bis* (2-chloroethylthio) ethane (Q)	4.7	6.0	3.4	0.09	0.13	0.07	1,4-dithioniabicyclo[2.2.2.] octane dichloride
2-(2-chloroethythio) ethyl 2-chloroethyl ether	5.3	6.4	3.8	4.6	5.6	3.0	S-(2-chloroethyl)-1,4-dithianium chloride
1,2-Dichloroethane	0.7	1.0	0.3	2.6	3.4	1.6	S-(2-hydroxyethyl)-1,4-dithianium chloride
1,4-Dithiane	1.5	2.0	1.4				Bis[2-(1,4-oxathianium)-S-ethyl] ether[2]
1,4-Thioxane	0.5	0.5	0.4	90	93	87	2-(1,4-dithianium)-S-ethyl 2-(1,4-oxathianium)-S ethyl ether[2]
							Bis [2-91,4-dithianium)-S ethyl] ether[2]

Liquid HT Metal Content (mg/Kg)	Liquid HT Metals Concentrations			Paste-Like Heel Metals Concentrations			Paste-Like Heel Metal Content (mg/Kg)
	Ave	Max	Min	Ave	Max	Min	
Aluminum	0.68	0.93	0.25	1.02	1.1	0.86	Aluminum
Antimony	0.059	0.062	0.055	0.25	0.34	0.19	Antimony
Arsenic	0.77	1.90	0.24	2.3	2.8	1.6	Arsenic
Barium	0.030	0.062	0.014	NR	NR	NR	Barium
Beryllium	NR	NR	NR	0.024	0.028	0.021	Beryllium
Boron	NR	NR	NR	NR	NR	NR	Boron
Cadmium	0.048	0.084	0.033	4.6	5.4	4.1	Cadmium
Chromium	0.24	0.27	0.23	15	23	5.8	Chromium
Cobalt	NR	NR	NR	1.8	1.9	1.7	Cobalt
Copper	1.9	3.5	1.0	120	140	110	Copper
Lead	0.37	0.54	0.15	1210	3100	130	Lead
Manganese	0.080	0.320	0.009	670	730	630	Manganese
Mercury	0.020	0.023	0.018	197	210	180	Mercury
Nickel	0.128	0.230	0.010	4.0	4.6	3.3	Nickel
Selenium	0.085	0.310	0.027	0.13	0.20	0.024	Selenium
Silver	0.081	0.140	0.047	0.092	0.13	0.053	Silver
Thallium	NR	NR	NR	NR	NR	NR	Thallium
Tin	0.070	0.150	0.050	4.7	5.8	4.2	Tin
Vanadium	0.12	0.18	0.09	0.11	0.13	0.10	Vanadium
Zinc	16	18	14	203	220	180	Zinc

[1]Organic Analysis by Nuclear Magnetic Resonance (NMR)

[2]Concentration of these ethers is determined as 100% minus the sum of the analytes that are ethers based on comments made in PWS Report by NMR analyst and not through actual analysis.

[3]Source is Table 6-33 of Summary of Parsons Engineering Design Study I Projectile Washout System (PWS) Testing, Vol. 1 of 3, 14 August 2004.

[4]Source is Table 6-37 of Summary of Parsons Engineering Design Study I Projectile Washout System (PWS) Testing, Vol. 1 of 3, 14 August 2004.

NR = Not Reported

Each of the metals analytical results for the three paste-like solids samples show a mercury (Hg) concentration of approximately 200 parts per million (ppm). The presence of the Hg is not attributed to cross-contamination by Lewisite because:

- The liquid HT metals results for all samples analyzed show no Hg present; the average liquid HT Hg concentration is 0.02 ppm. The average liquid HT arsenic (As) concentration was 0.77 ppm. Past incidences of Hg contamination caused by Lewisite have resulted in both Hg and As concentrations measuring in hundreds to thousands of parts per million, far above those concentrations found in the liquid HT.
- The paste-like substance metals results for all samples show elevated concentrations of Hg; however, the average concentration of As was not also elevated as would be expected with Lewisite cross-contamination. The As concentration in the paste-like solids averaged 2.3 ppm.
- Both a liquid and a paste-like solid sample were collected from the same mortar round. The results showed the Hg to be elevated in the solid sample only. Neither sample had elevated concentrations of As. [Past results for other agents that were contaminated from being transferred to ton containers that had previously held Lewisite had analytical results showing elevated concentrations of Hg and As in both the liquid and solid fractions (see Table 3)].

Table 3: Parsons PWS Liquid HT and Solid Heel Analysis for Samples Taken from Same Mortar

Sample ID	PBCD04PT1ABX[1]	PBCD03PT01CX[2]	Sample ID
Sample Description	HT from Mortar	Solids from Mortar	Sample Description
Single or Mixed	Single	Single	Single or Mixed
Lot #	**500-6**	**500-6**	Lot #
Solids Present	Yes	Yes	Solids Present
Mortar Number	**26**	**26**	Mortar Number
Organic Compound	**(wt%)**	**Organic Compound**	**(wt%)**
Bis (2-chloroethyl) sulfide (HD Agent)	52.3	0.44	1,4-Dithiane
bis [2-(2-chloroethylthio)ethyl] ether (T)	34.57	0.84	1,4-Thioxane
1,2-*bis* (2-chloroethylthio) ethane (Q)	5.02	0.13	1,4-dithioniabicyclo[2.2.2.] octane dichloride
2-(2-chloroethythio) ethyl 2-chloroethyl ether	5.58	3.0	S-(2-chloroethyl)-1,4-dithianium chloride
1,2-Dichloroethane	0.94	2.8	S-(2-hydroxyethyl)-1,4-dithianium chloride
1,4-Dithiane	1.42		Bis[2-(1,4-oxathianium)-S-ethyl] ether
1,4-Thioxane	0.35	93	2-(1,4-dithianium)S-ethyl 2-(1,4-oxathianium)-S-ethyl ether
			Bis[2-91,4-dithianium)-S-ethyl] Ether
Mass Balance	100.18	100	Mass Balance
Liquid HT Metal Content (mg/Kg)		**Paste-Like Solids HT Metal Content (mg/Kg)**	
Aluminum	0.71	0.86	Aluminum
Antimony	0.061	0.34	Antimony
Arsenic	0.64	2.8	Arsenic
Barium	0.024	NR	Barium
Beryllium	NR	0.028	Beryllium
Boron	NR	NR	Boron
Cadmium	0.033	5.4	Cadmium
Chromium	0.26	5.8	Chromium
Cobalt	NR	1.8	Cobalt
Copper	2.3	140	Copper
Lead	0.26	130	Lead
Manganese	0.048	630	Manganese
Mercury	0.02	200	Mercury
Nickel	0.046	4.1	Nickel
Selenium	0.092	0.024	Selenium
Silver	0.047	0.053	Silver
Thallium	NR	NR	Thallium
Tin	0.056	5.8	Tin
Vanadium	0.12	0.10	Vanadium
Zinc	16	180	Zinc

[1]Source is Table 6-33 of Summary of Parsons Engineering Design Study I Projectile Washout System (PWS) Testing, Vol. 1 of 3, 14 August 2004.

[2]Source is Table 6-37 of Summary of Parsons Engineering Design Study I Projectile Washout System (PWS) Testing, Vol. 1 of 3, 14 August 2004.

NR = Not Reported

1.2 PROJECT OBJECTIVES

The main objective of this S&A plan is to characterize the DCD 4.2-inch HT Mortar stockpile to ensure that the planned feed rates of mortars to the MPF and liquid HT to the Liquid Incinerators (LICs) result in operations that are compliant with the performance standards and feed rate limits specified in the TOCDF RCRA Permit. In addition, this plan seeks to provide the collection of additional data for waste characterization in a manner that does not jeopardize the safety of the work force or endanger the environment. These safety and environmental goals must be maintained throughout the sampling and testing process.

This plan is designed to meet the following objectives for characterization of HT stored in 4.2-inch HT Mortars at DCD:

- Determine organic compound concentrations in the liquid HT samples using a gas chromatograph equipped with a mass spectrometer and a flame ionization detector (GC/MS/FID).
- Determine the presence of Tentatively Identified Compounds (TICs) in HT by GC/MS/FID. Peaks in the GC/MS chromatogram that are at least 1.0 percent of the total area will be identified as TICs, and a total summation of TICs will be reported.
- Analyze liquid HT samples for the analytes specified in Table 4.

Table 4: HT Organic Analytes

HT Analytes	Analysis Type
Bis (2-chloroethyl) sulfide (HD Agent)	Quantitative, through use of calibration standard
bis [2-(2-chloroethylthio)ethyl] ether (T)	Semi-quantitative, identifed as a TIC with concentration estimated from area under response curve (calbration standard not used)
1,2-*bis* (2-chloroethylthio) ethane (Q)	
2-(2-chloroethythio) ethyl 2-chloroethyl ether	
1,2-Dichloroethane	
1,4-Dithiane	
1,4-Thioxane	

- Analyze the ACS Tank liquid HT samples for chlorides using Tooele Laboratory Operating Procedure (TEP-LOP)-584 (bomb calorimeter), followed by offsite analysis by SW-846, Method 9056.
- Analyze liquid HT and paste-like solid samples for total metals using an Inductively Coupled Plasma/Mass Spectrometer (ICP/MS); the metals of interest are: aluminum (Al), antimony (Sb), arsenic (As), barium (Ba), beryllium (Be), boron (B), cadmium (Cd), chromium (Cr), cobalt (Co), copper (Cu), lead (Pb), manganese (Mn), mercury

(Hg), nickel (Ni), selenium (Se), silver (Ag), tin (Sn), thallium (Tl), vanadium (V), and zinc (Zn).

The 4.2-inch HT Mortars will be drained prior to being fed to the Metal Parts Furnace (MPF). The drained liquid HT will be collected in the Agent Collection System (ACS) Tanks and fed to the LICs. Two separate matrices will be sampled: Liquid HT samples will be collected from one of the ACS Tanks; after draining, and with a residual 15 percent liquid HT heel remaining, a subset of the drained mortar casings will be assayed to determine the frequency of occurrence of the paste-like solid heel. Samples will be collected from a subset of the mortars found to contain paste-like solid heels.

The liquid HT samples taken from the ACS Tank will be analyzed for the organic compounds shown in Table 4, and for the metals used in the DCD Human Health Risk Assessment (HHRA). The paste-like solid samples and liquid HT samples collected from individual mortars will be analyzed for HHRA metals. The analyses will be conducted at the TOCDF Chemical Assessment Laboratory (CAL).

1.3 PLAN ASSUMPTIONS

This plan is based on the following assumptions:

- Three liquid HT samples will be collected from an ACS Tank during three separate sampling events; each of the three samples will represent a composite of agent drained from approximately 670 mortars.
- No more than 112 drained HT mortars will be assayed for the presence of paste-like solid heels.
- Up to three samples of paste-like solids will be collected from each of three drained mortars found to have solids.
- Up to three liquid HT samples will be collected from the same mortars from which paste-like solid heels are collected.
- The samples will be properly labeled, and all chain-of-custody (COC) paperwork will be maintained.
- The samples will be prepared and analyzed at the CAL.
- Reports of analytical results will be prepared and submitted to DSHW by EG&G as data are generated.
- LIC processing will resume once the samples have been collected. The LIC HT feed rate will be reduced to 90 percent of the permitted limit until results of the chloride analysis are received and submitted to DSHW. Processing mortars in the MPF will also resume; the metal of concern for the MPF is Hg, and the MPF exhaust gas is continuously sampled. The resulting samples will be analyzed for Hg per the requirements of an

Alternative Monitoring Request, which was approved by the EPA and administered by the State of Utah Division of Air Quality (DAQ).

- Liquid HT samples will continue to be collected from the ACS Tanks on a weekly basis after completion of the sampling plan. No further sampling for the paste-like solid heel will occur after completion of this plan.

2.0 SAMPLING METHODS

2.1 LIQUID HT SAMPLE COLLECTION

There are 62,524 4.2-inch HT Mortars in storage at DCD, divided into seven lots. As previously discussed, each of the DCD lots contains a mixture of mortars from the initial PBA manufacturing lot number since the mortars have undergo two renovations, and with each renovation, they were assigned new lot numbers. Further, because the Army did not consider the HT fill of the mortars to be a critical component, no HT agent manufacturing lot numbers were recorded. The lack of additional information necessitates considering these 62,524 4.2-inch HT Mortars a single population. Table 5 shows the number of mortars in each DCD lot.

Table 5: DCD 4.2" HT Mortar Inventory

DCD Lot Number	No. in Lot
TOD-500-1	12,970
TOD-500-2	10,579
TOD-500-3	7,461
TOD-500-4	8,410
TOD-500-5	12,385
TOD-500-6	10,621
TOD-500-7	98
Total ⇒	62,524
Total Excluding TOD-500-1 ⇒	49,554

A review of records has been conducted concerning the manufacture, renovation, and movement of the national 4.2-inch HT Mortar stockpile. From this information, along with some assumptions, it is possible to estimate the distribution of PBA agent fill lots in the DCD assembly lots. Table 6 shows the PBA agent fill lot distribution and number of mortars per lot that are assigned to each DCD lot. (Note that information was not available for lot TOD-500-1.)

Table 6: PBA Fill Lot Distribution in DCD 4.2" HT Mortar Stockpile

DCD Assembly Lot Number	PBA Manufacturing Lot Number	No./PBA Lot	No./TOD Lot	DCD Assembly Lot Number	PBA Manufacturing Lot Number	No./PBA Lot	No./TOD Lot
TOD-500-2	PBA-143-91	1		TOD-500-5	PBA-143-90	154	
TOD-500-2	PBA-143-95	3,602		TOD-500-5	PBA-143-97	3	
TOD-500-2	PBA-143-96	2		TOD-500-5	PBA-143-98	2,528	
TOD-500-2	PBA-143-101	3,516		TOD-500-5	PBA-143-99	152	
TOD-500-2	PBA-143-106	3,500	10,621	TOD-500-5	PBA-143-100	39	
TOD-500-3	PBA-143-66	3,189		TOD-500-5	PBA-143-103	1,436	
TOD-500-3	PBA-143-70	1,949		TOD-500-5	PBA-143-104	1,678	
TOD-500-3	PBA-143-75	1,412		TOD-500-5	PBA-143-105	3	12,427
TOD-500-3	PBA-143-94	907		TOD-500-6	PBA-143-66	5	
TOD-500-3	PBA-143-95	1		TOD-500-6	PBA-143-67	114	
TOD-500-3	PBA-143-106	3	7,461	TOD-500-6	PBA-143-70	6	
TOD-500-4	PBA-143-58	2		TOD-500-6	PBA-143-75	19	
TOD-500-4	PBA-143-66	2		TOD-500-6	PBA-143-91	3,536	
TOD-500-4	PBA-143-68	2		TOD-500-6	PBA-143-96	3,525	
TOD-500-4	PBA-143-69	2,777		TOD-500-6	PBA-143-99	4	
TOD-500-4	PBA-143-71	908		TOD-500-6	PBA-143-102	3,491	10,700
TOD-500-4	PBA-143-72	1,936		TOD-500-7	PBA-143-28	6	
TOD-500-4	PBA-143-73	1,954		TOD-500-7	PBA-143-66	2	
TOD-500-4	PBA-143-74	803		TOD-500-7	PBA-143-67	3	
TOD-500-4	PBA-143-91	1		TOD-500-7	PBA-143-68	5	
TOD-500-4	PBA-143-97	24		TOD-500-7	PBA-143-69	10	
TOD-500-4	PBA-143-100	42		TOD-500-7	PBA-143-71	3	
TOD-500-4	PBA-143-105	2	8,453	TOD-500-7	PBA-143-74	1	
TOD-500-5	PBA-143-54	2		TOD-500-7	PBA-143-91	2	
TOD-500-5	PBA-143-58	8		TOD-500-7	PBA-143-95	4	
TOD-500-5	PBA-143-67	335		TOD-500-7	PBA-143-98	12	
TOD-500-5	PBA-143-68	3,229		TOD-500-7	PBA-143-99	18	
TOD-500-5	PBA-143-69	2		TOD-500-7	PBA-143-100	6	
TOD-500-5	PBA-143-71	1,741		TOD-500-7	PBA-143-101	1	
TOD-500-5	PBA-143-72	3		TOD-500-7	PBA-143-103	16	
TOD-500-5	PBA-143-73	2		TOD-500-7	PBA-143-104	7	96
TOD-500-5	PBA-143-74	1,112		**Total Excluding DCD Lot TOD-500-1⇒**			**49,758**

Note: Total per DCD Lot presented in Tables 5 and 6 do not match. Difference is attributed to the mortars expended over the years to testing, and inspections.

The liquid HT samples will be collected from ACS-Tank-101, which has a 500-gallon working volume. The HT mortars will be drained of their agent fill. An approximate 15 percent by weight liquid heel will be left in each drained mortar to prevent plugging the drain probe should the mortar being drained contain a paste-like solid heel.

There is sufficient space in the Munitions Processing Bay (MPB) and Lower Buffer Storage Area (LBSA) to stage seven full trays of 96 drained HT mortars. Seven trays of HT mortars will be prepared; each tray will be loaded with mortars from a different DCD assembly lot. On a single tray, each mortar will be from the same DCD assembly lot. Considering the information provided in Table 6 and the probability of selecting a particular mortar from a particular lot, the mix of agent in the receiving ACS Tank will represent all but six of the original 28 PBA manufacturing lots that are thought to comprise the DCD stockpile, with the contribution of agent from each of these lots averaging about 3 percent of the ACS Tanks fills. The samples collected from the ACS Tanks, therefore, provide a representative sample of the HT filling the DCD 4.2-inch Mortars, and Table 7 provides the likely distribution of liquid versus solid distribution for these munitions.

Table 7: Likely distribution of Liquid HT in ACS-Tank Based on No. of Mortar per Lot

DCD Assembly Lot No.⇒ / PBA Manufacturing Lot No.	TOD-500-7 (no.mortars)	TOD-500-6 (no.mortars)	TOD-500-5 (no.mortars)	TOD-500-4 (no.mortars)	TOD-500-3 (no.mortars)	TOD-500-2 (no.mortars)	TOD-500-1 (no.mortars)	Total Mortar from Lots[1]	% of Tank Vol.
Not Identified							12.970	96	
PBA-143-106					3	**3,500**		32	5%
PBA-143-105			3	2				0	0%
PBA-143-104	7		**1,678**					20	3%
PBA-143-103	16		**1,436**					27	4%
PBA-143-102		**3,491**						31	5%
PBA-143-101	1					**3,516**		33	5%
PBA-143-100	6		39	42				6	1%
PBA-143-99	18	4	152					18	3%
PBA-143-98	12		**2,528**					32	5%
PBA-143-97			3	24				0	0%
PBA-143-96		**3,525**				2		32	5%
PBA-143-95	4				1	**3,602**		37	6%
PBA-143-94					**907**			12	2%
PBA-143-91	2	**3,536**		1		1		34	5%
PBA-143-90			154					0	0%
PBA-143-75		19			**1,412**			18	3%
PBA-143-74	1		**1,112**	**803**				19	3%
PBA-143-73			2	**1,954**				22	3%
PBA-143-72			3	**1,936**				22	3%
PBA-143-71	3		**1,741**	**908**				27	4%
PBA-143-70		6			**1,949**			25	4%
PBA-143-69	10		2	**2,777**				42	6%
PBA-143-68	5		**3,229**	2				30	5%
PBA-143-67	3	114	335					3	0%
PBA-143-66	2	5		2	**3,189**			43	6%
PBA-143-58			8	2				0	0%
PBA-143-54			2					0	0%
PBA-143-28	6							6	1%
total per lot ⇒	96	10,700	12,427	8,453	7,461	10,621	12.970	ave	3%
								max	6%
								min	1%

[1]Total of mortar from each PBA lot most likely to be present on 7 tray of mortars selected by each DCD (i.e., TOC) lot.
Note: Bolded value represent PBA lots most likely to be represented in ACS-Tank sample because of number of mortars per lot relative to the total number of mortars comprising the DCD lot. Information was not available for lot TOC-500-1.

Three samples of liquid HT will be collected from ACS-Tank-101 after the contents from the mortars on the first seven trays have been drained and transferred to the tank. The ACS-Tank-101 was selected because its smaller volume will lend itself to more effective circulation of the contents. This consideration is important since these samples will be collected at the beginning of the 4.2-inch HT Mortar Campaign while processing rates are being "ramped-up" and processing issues associated with the demilitarization machines are being resolved. Additionally, feed rate to the MPF will also require a "ramp-up." As planned, samples can be collected after the processing of seven trays (or 672 mortars). The ACS-Tank-102 would take over 2,400 mortars, or about 25 trays, to fill; the ACS-Tank-101 will only take about 11 trays or mortars. However, during the "ramp-up" period, it is difficult to predict the time required to process a specified number of items.

Seven trays of HT mortars drained to 15 percent heels represent about:

$$7(\text{trays/mortars}) * 96(\text{mortar/tray}) * 5.8(\text{lb HT/mortar}) * (1 - 0.15) * 1((\text{gal}/(1.27 * 8.34\ \text{lb})) = 312\ \text{gal of HT}$$

This amount of agent will result in this tank being about half full. The tank's contents will be circulated for at least 0.5 hr before the sample is collected, which for this amount of agent represents about two tank "turn overs" based on the pump size and eight "turn overs" based on the eductor, which is designed to give a 4:1 mixing ratio. Once the sample is collected, the contents of the tank will be processed in one of the LICs to make room for the next agent sampling event.

The second liquid HT sample will also be collected from ACS-Tank-101. The tank will be filled with the agent drained from at least seven trays of mortars. The tank's contents will be circulated for at least 0.5 hr before the sample is collected. Once the sample is collected, the contents of the tank will be processed in one of the LICs.

The third liquid HT sample will again be collected from ACS-Tank-101. A duplicate sample of the liquid HT matrix will additionally be collected during the third ACS Tank sampling event. The tank will be filled with the agent drained from at least seven trays of mortars, and the contents will be circulated for at least 0.5 hr before the sample is collected. Once the sample is collected, the contents of the tank will be processed in one of the LICs.

Note that the contents of all but two of the mortars comprising TOD-500-7 will be included in the first liquid HT sample collected from the ACS Tank. Therefore, the seventh tray associated with the second and third ACS Tank samples will be comprised of mortars from any of the six remaining TOD lot numbers.

Sample batch sizes of 312 gal of liquid HT are selected to allow the samples to be collected in a reasonable time frame (ideally, over a three day period).

2.2 PASTE-LIKE SOLID AND LIQUID SAMPLE COLLECTION

During the PWS tests conducted by Parsons Corporation, it was determined that some of the DCD HT mortars contained a paste-like solid that had settled to the bottom of the mortars. The analysis of these solids showed them to contain Hg at concentrations of approximately 200 ppm. The rate of occurrence of these solids for the lot tested was about one in three. This was thought perhaps to be a lot-specific anomaly until the information about the relationship between the PBA fill lots and the DCD assembly lots became available. The results for the single mortar round from which both a liquid and solid sample were taken showed Hg concentrations in the liquid HT of about 0.02 ppm, and solid concentrations of about 200 ppm (see Table 3).

To develop an estimate of the occurrence of the paste-like solid heel, TOCDF will assay each drained mortar in the outer most row of each tray. The 4.2-inch Mortars are placed on the MPF burn trays in an array that is six mortars wide and sixteen mortars long. For ease of access, the drained mortars in the outer most rows are selected for assay. Sixteen mortars on each tray will be available for assay (i.e., the number of mortars in a single file/column), and a total of 112 mortars will be assayed. Solid samples will be collected from three of the mortars found to contain paste-like solid heels. A liquid HT sample will be collected from each of these same three mortars. The results will be used to confirm that the presence of Hg is limited to the paste-like solid heel, which will be processed in the MPF, and is not contained at concentrations of concern in the liquid HT that is removed from mortars having paste-like heels. If these results conform to those obtained during the Parsons Corporation PWS test (i.e, no mercury in the liquid) then there will be no Hg concerns about feeding HT drained from mortars with solid heels to the LICs. The paste-like heels are expected to contain mercury, which is an MPF issue that is mitigated by the continuous MPF Hg exhaust gas monitoring.

The assay of the HT mortars, and the associated collection of solid and liquid samples from three of the mortars found to contain the paste-like solids, will occur when the first seven trays of drained mortars are available. In addition, duplicate liquid HT and paste-like solid samples will be collected from one of the three mortars sampled.

If no paste-like solids are encountered during the first day of sampling, the second set of seven trays will be assayed as previously described. If no paste-like solids are encountered during the second attempt, no additional attempts will be made to collect paste-like solid samples.

2.2 SAMPLE COLLECTION METHODS

The mortars are moved from their storage site at DCD Area 10 to the TOCDF Container Handling Building (CHB) in On-Site Containers (ONCs). The ONCs are stored in the CHB until they are moved into the Munitions Demilitarization Building (MDB) where the munitions are removed from the ONCs and then processed. Once the mortar fuzes and bursters are removed by Projectile/Mortar Disassembly Machines (PMDs) in one of the Explosive Containment Rooms

(ECR), the mortars are transferred to the Munitions Processing Bay (MPB) where their burster wells are removed and their liquid HT fill is drained by one of three Multipurpose Demilitarization Machines (MDMs). The liquid HT will be transferred to ACS-Tank-101, selected for its smaller volume, which will lend itself to more effective circulation of the contents. The liquid HT samples from ACS-Tank-101 will be collected from a tap located on the recirculation return line.

The assay for the occurrence of the paste-like solid heels and sample collection of heels and liquid HT from drained mortars will occur in the LBSA. The mortars in this location will have been drained to contain an approximate 15 percent heel. Each mortar on one of the outside rows (each row consisting of 16 mortars) of each of seven trays will be assayed for the occurrence of paste-like solid heels. Samples will be collected from three of the mortars found to contain the solid heels. A liquid sample will be collected from each of the same three mortars from which the solid heel samples are collected. A duplicate solid heel and liquid sample will be collected from one of those mortars sampled. The tools that will be used to collect these samples have not yet been determined.

The 1-mL liquid HT samples will be placed in separate vials and transferred to the CAL for analysis. Each solid sample will be approximately 1 gram. These samples will be transferred to separate vials and sent to the CAL for metals analyses.

3.0 ANALYTICAL METHODS

Liquid HT samples collected from ACS-Tank-101 are evaluated for organic compounds, HHRA metals, and chlorine. Liquid HT samples collected from the mortars are evaluated for HHRA metals.

3.1 LIQUID MUSTARD ORGANIC COMPOUND ANALYSES

The organic compounds are analyzed by the CAL using the method specified in Tooele Laboratory Operating Procedure 584 (TE-LOP-584). Sample aliquots are weighed and then diluted with isopropyl alcohol to a known volume. The diluted samples are analyzed as specified in the above reference procedure. The *bis*(2-chloroethyl) sulfide that is present is quantitated using a standard. The other analytes listed below will be reported as TICs based on peaks in the chromatogram that are at least 1.0 percent of the total area.

- *bis*(2-chloroethyl) sulfide (mustard);
- 1,2-*bis*(2-chloroethylthio)ethane (Q);
- *bis*[2-2-(chloroethylthio)ethyl]ether (T);
- 2-(2-chloroethythio) ethyl 2-chloroethyl ether;
- 1,2-dichloroethane;
- 1,4-dithiane; and
- 1,4-thioxane

3.2 TOTAL METALS ANALYSES

All liquid HT samples and the paste-like solid heel samples will be analyzed for total HHRA metals by ICP/MS using TE-LOP-557. The agent samples are prepared for analysis using TE-LOP-584 by digesting an aliquot of mustard in a combination of hydrochloric acid and nitric acid, and then heating it in a microwave oven. The heel samples will be digested using a similar microwave digestion procedure. The digested samples are then diluted to a known volume and analyzed by aspirating the solution into the plasma, which produces an atomic vapor. The mass spectrometer separates the elements by their mass, and these elements are quantitated against internal standards. The metals analyzed are Al, Sb, As, Ba, Be, B, Cd, Cr, Co, Cu, Pb, Mn, Hg, Ni, Se, Ag, Tl, Sn, V, and Zn.

3.3 TOTAL CHLORINE

Total chlorine analyses are proposed for the liquid HT samples collected from ACS-Tank-101. These results will be used to ensure that the LIC chlorine feed rate limit is not exceed during the

4.2-inch HT Mortar Campaign. The LIC chlorine feed rate limit was established by feeding distilled *bis*(2-chloroethyl) sulfide (HD). It is anticipated that the chlorine content of HT will be less than that of HD because of the larger molecules comprising the HT mixture (i.e., molecules contain more carbon, hydrogen, and sulfur atoms than does HD). However, to ensure compliance with the LIC chlorine feed rate, TOCDF will limit the feed rate of HT fed to the LICs to 90 percent of the permitted limit (i.e., 0.9 * 1,208 = 1,087 lb/hr) until results are available and have been reviewed and approved by the DSHW. (Note that the chlorine analysis is conducted offsite.)

The MPF chlorine feed rate was reestablished during the MPF 155-mm (155) Projectile Mustard (H) Agent Trial Burn (ATB) while feeding full (undrained) projectiles. Because the 4.2-inch HT Mortars will be drained of most of their agent fill, there is no possibility of exceeding the chlorine feed rate to the MPF. Therefore, the samples collected from the mortars will not be analyzed for chlorine.

Table 8 provides a summary of the planned sampling and analysis.

Table 8: Sample & Analyisis Summary

Sample Source	Sample Matrix	No. Samples	Analytical Parameters	Sample Prep/ Analysis Methods
ACS-Tank-101	Liquid	4, including one duplicate	•Organic Compounds per Table 4	TE-LOP-584
			•HHRA Metals	TE-LOP-557
			•Chlorides	TE-LOP-584/ SW-846 M9056
Drained Mortar w/ Solid Heel Stored in LBSA	Liquid	4, including one duplicate	•HHRA Metals	TE-LOP-557
Drained Mortar w/ Solid Heel Stored in LBSA	Solid	4, including one duplicate	•HHRA Metals	TE-LOP-557

4.0 QUALITY ASSURANCE/QUALITY CONTROL OBJECTIVES

The overall objective for the data collected during this project is to establish concentration ranges for metals and organic compounds. The analytical data will be used to characterize the HT in the 4.2-inch Mortars. The collected samples will be analyzed by the CAL using standard methods. The organic compounds will be analyzed by TE-LOP-584, while the metals analyses will be conducted in accordance with TE-LOP-557. To assess the quality of the data collected during this project, a series of Data Quality Objectives (DQOs) have been set for each method used for sample analyses. Summaries of the DQOs for the analytical data are presented in Tables 4 and 5. The DQOs will be evaluated against the data acceptance criteria listed in the tables. The DQOs identify the target precision and accuracy limits that are used to assess the data quality. These two tables were developed using the criteria in SW-846 (1), EPA QA/G-5 (3), and in the QA/QC Handbook (4).

The field and analytical data will be reviewed, and a complete assessment of the data quality indicators will be included in the information provided to DSHW.

Several procedures will be used for monitoring the precision and accuracy objectives of the analytical program. Calibration standards, internal standards, and laboratory standards (QL) will be of high-purity materials, when available. Standards of chemical agent materials will be obtained from the Army. Analytical instruments will be calibrated per TE-LOP-584 requirements prior to sample analysis to demonstrate that accurate performance levels are being met. Data precision and accuracy will be assessed by evaluating the results of the analyses of internal standards, surrogate compounds, laboratory blanks, calibration check standards, reagent blanks, method blanks, duplicate samples, and matrix or surrogate spiked samples.

When analytical QC procedures reveal that a measurement error has exceeded the target criterion, the source of the deviation will be identified, and corrective action will be taken (as described in the tables); those data points will be flagged and discussed with the DSHW. Alternative procedures (either sampling or analytical) will be considered and recommended to the analytical project manager when necessary. Any changes or additions will be discussed with the DSHW prior to implementation.

TABLE 9. Summary of Data Quality Objectives for Organic Compounds

NOTE: This table will be revised based on the DSHW approved version of TE-LOP-584.

Table 10. Summary of Data Quality Objectives for ICP/MS Analyses (TE-LOP-557)

QUALITY PARAMETER	METHOD/ FREQUENCY	CRITERIA	CORRECTIVE ACTION
Instrument Tune	Daily, prior to calibration and sample analysis	Mass resolution < 1.0 amu @ 10 % peak height Mass calibration ± 0.1 amu	Retune instrument; Repeat tune performance verification
Initial Calibration	Blank and at least one standard	ICV ± 10 % of expected value	Evaluate and reanalyze ICV; Recalibrate
Calibration Blank	After ICV and CCV	< PQL	Clean system; Rerun; Reanalyze affected samples
CCV	Every 10 samples and end-of-run sequence	± 10 % of expected value	Reanalyze CCV; Recalibrate; Reanalyze samples
Method Blank	1 per analytical batch	<PQL	Reanalyze; Recalibrate as necessary
Internal Standard	Each sample	30-200 %R	Reanalyze and/or narrate
Laboratory Control Sample (LCS)	1 per analytical batch	75 to 125 %R	Check calculations; Assess impact on data; Re-digest and reanalyze as necessary; Narrate
MS/MSD	1 set per analytical batch	75-125 %R – RPD ≤ 20 % - Aqueous 50-150 %R – RPD ≤ 50% - Solids	Assess impact on data; Narrate
Duplicate Analyses	1 per analytical batch	RPD ≤ 20 % - Aqueous RPD ≤ 50 % - Solids	Check calculations; Reanalyze; Assess impact on data
Holding Time		30 days to analysis	

Notes:

amu = atomic mass unit
ICV = Initial Calibration Verification
CCV = Continuing Calibration Verification

Table 11. Summary of Data Quality Objectives for Chlorine Analyses of Trial Burn Samples (SW-846 Method 9056)

QUALITY PARAMETER	METHOD/ FREQUENCY	CRITERIA	CORRECTIVE ACTION
Method Blank	1 per analytical batch	< PQL	Reanalyze; Assess impact on data; Narrate
Initial Calibration	4-point calibration and a blank. Initial/as required	Correlation coefficient > 0.995	Evaluate system; Recalibrate
Continuing Calibration	Midpoint standard every 10 samples and at end of sequence	90 – 110 %R	Evaluate system; Repeat calibration check; Recalibrate; Reanalyze affected samples
Precision/ Accuracy	LCS per batch	90 – 110 %R	Check calculations; Reanalyze; Assess impact on data; Narrate
	MS/MSD per batch	85 – 115% %R, RPD ≤ 25 %	Check calculations; If RPD is in control, accept data and narrate; If RPD is out of control, reanalyze.
Audit Sample	As provided	90 – 110 %R	Check calculations; Reanalyze and Narrate;
PQL	Chlorine	< 0.1 Wt%	
Holding Time		28 days	

Note:
The term PQL refers to the laboratory's standard Reporting Limit.

5.0 REFERENCES

(1) *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, 3rd Edition including Update III, USEPA, SW-846, December 1996.

(2) *HQ and HT, Review of British and U.S. Literature*, N.W. Cone, C.A. Rouller, February 1943.

(3) *EPA Guidance for Quality Assurance Project Plans*, EPA QA/G-5, December 2002.

(4) *Handbook: Quality Assurance/Quality Control (QA/QC) Procedures for Hazardous Waste Incineration*, EPA/625/6-89/023, January 1990.